

Claims

- [c1] 1. A process for reduction and/or removal of permanganate-reducing compounds (PRC's) and C_{2-12} alkyl iodide compounds formed in the carbonylation of a carbonylatable reactant selected from the group consisting of methanol, methyl acetate, methyl formate and dimethyl ether and mixtures thereof to produce a product comprising acetic acid, comprising the steps of: separating said carbonylation product to provide a volatile phase comprising acetic acid, and a less volatile phase; distilling said volatile phase to yield a purified acetic acid product and a first overhead comprising organic iodide, water, acetic acid, and at least one PRC; distilling at least a portion of the first overhead in a distillation apparatus to form a PRC enriched second overhead; extracting the second overhead with water and separating therefrom an aqueous stream comprising said at least one PRC; and recycling at least a first portion of the extracted second overhead to said distillation apparatus.

- [c2] 2. The process of claim 1, wherein the improvement further comprises introducing at least a second portion of the extracted second overhead directly or indirectly into the reaction medium.
- [c3] 3. The process of claim 2, wherein said second overhead comprises dimethyl ether in an amount effective to reduce the solubility of methyl iodide in said aqueous stream.
- [c4] 4. The process of claim 2, further comprising the step of adding dimethyl ether to the second overhead.
- [c5] 5. The process of claim 2, further comprising forming dimethyl ether in said distillation apparatus.
- [c6] 6. The process of claim 5, further comprising the step of adding water to a stream associated with said distillation apparatus, whereby dimethyl ether is formed in the distillation apparatus.
- [c7] 7. The process of claim 1, wherein the recycled first portion of the extracted second overhead is fed to the distillation apparatus together with the first overhead portion.
- [c8] 8. The process of claim 1, wherein the recycled first portion of the extracted second overhead is fed to the dis-

tillation apparatus separately from the first overhead portion.

- [c9] 9.The process of claim 1, further comprising the step of adding dimethyl ether into at least one stream selected from the group consisting of said volatile phase, said first overhead, said second overhead, a reflux stream associated with the distillation of said volatile phase, and a reflux stream of said distillation apparatus.
- [c10] 10.The process of claim 1, wherein said at least one PRC comprises acetaldehyde.
- [c11] 11.The process of claim 10, wherein a sufficient quantity of said acetaldehyde is removed from said volatile phase to maintain a concentration of less than about 400 parts per million by weight of propionic acid in said purified acetic acid product.
- [c12] 12.The process of claim 10, wherein a sufficient quantity of said acetaldehyde is removed from said volatile phase to maintain a concentration of less than about 250 parts per million by weight of propionic acid in said purified acetic acid product.
- [c13] 13.The process of claim 1, wherein the step of distilling said first overhead comprises a plurality of consecutive distillation steps, and wherein the first portion of said

extracted second overhead is recycled to a stream associated with the second or later step of said distillation steps.

- [c14] 14.A process for producing acetic acid, comprising the steps of:
- (a) carbonylating at least one reactant selected from the group consisting of methanol, methyl acetate, methyl formate and dimethyl ether in a reactor containing a suitable reaction medium;
 - (b) separating the products of said carbonylation into a volatile product phase comprising acetic acid and at least one permanganate reducing compound (PRC), and a less volatile phase;
 - (c) distilling said volatile product phase to yield a purified acetic acid product and a first overhead comprising organic iodide, water, acetic acid, and said at least one PRC;
 - (d) distilling at least a portion of the first overhead to produce a PRC-enriched second overhead; and
 - (e) extracting the second overhead with water and separating therefrom an aqueous extract containing concentrated PRC's for disposal,
- wherein at least a first portion of the extracted second overhead is recycled and distilled in step (d) with the first overhead.

- [c15] 15. The process of claim 14, wherein said second overhead comprises dimethyl ether in an amount effective to reduce the solubility of methyl iodide in said aqueous stream.
- [c16] 16. The process of claim 14, further comprising the step of adding dimethyl ether to at least one stream selected from the group consisting of said volatile phase, said first overhead, said second overhead, a reflux stream associated with the distillation of the volatile phase, and a stream associated with the distillation of the first overhead portion.
- [c17] 17. The process of claim 14, further comprising recycling at least a second portion of the extracted second overhead directly or indirectly to the reactor.
- [c18] 18. The process of claim 17, further comprising forming dimethyl ether during the distillation of the first overhead portion and reacting at least a portion of the dimethyl ether with carbon monoxide in the reactor.
- [c19] 19. The process of claim 18, further comprising the step of injecting water into the first overhead or into the first portion of the extracted second overhead to promote formation of dimethyl ether during the distillation of the first overhead portion.

- [c20] 20. The process of claim 14, wherein the step of distilling at least a portion of the first overhead comprises a plurality of consecutive distillation steps, and wherein the first portion of said extracted second overhead is recycled to a stream associated with the second or later step of said distillation steps.
- [c21] 21. The process of claim 14, wherein said at least one PRC comprises acetaldehyde.
- [c22] 22. The process of claim 21, wherein a sufficient quantity of said acetaldehyde is removed from said volatile phase to maintain a concentration of less than about 400 parts per million by weight of propionic acid in said purified acetic acid product.
- [c23] 23. The process of claim 21, wherein a sufficient quantity of said acetaldehyde is removed from said volatile phase to maintain a concentration of less than about 250 parts per million by weight of propionic acid in said purified acetic acid product.
- [c24] 24. A process for separating a mixture containing water, acetic acid, methyl iodide, methyl acetate, methanol, at least one C₂₋₁₂ alkyl iodide and at least one permanganate reducing compound (PRC), comprising:
(a) distilling the mixture to provide a PRC enriched over-

head stream comprising methyl iodide, water and said at least one PRC;

(b) extracting the PRC enriched overhead stream with water and separating therefrom an aqueous stream containing said at least one PRC; and

(c) distilling at least a first portion of the extracted PRC enriched overhead with the mixture.

[c25] 25.The process of claim 24, wherein the step of distilling the mixture comprises a plurality of consecutive distillation steps, and wherein the first portion of said extracted PRC enriched overhead is recycled to a stream associated with the second or later step of said distillation steps.

[c26] 26.The process of claim 24, further comprising the step of adding dimethyl ether to at least one stream selected from the group consisting of said mixture, said PRC enriched overhead, and streams associated with said distillation.

[c27] 27.The process of claim 24, wherein said second overhead comprises dimethyl ether in an amount effective to reduce the solubility of methyl iodide in said aqueous stream.

[c28] 28.The process of claim 24, further comprising the step of providing said mixture by separating a liquid compo-

sition into a light phase and a heavy phase, said liquid composition comprising water, acetic acid, methyl iodide, methyl acetate, methanol, at least one C₂₋₁₂ alkyl iodide and said at least one PRC, wherein the light phase comprises said mixture and the heavy phase comprises methyl iodide.

[c29] 29. The process of claim 28, further comprising the steps of:

performing a liquid-vapor phase separation on the effluent of a methanol carbonylation reactor to form a vapor phase and a liquid phase;

distilling the vapor phase to form a first overhead and a liquid product; and

condensing at least a portion of the first overhead to provide said liquid composition.

[c30] 30. The process of claim 29, wherein said at least one PRC comprises acetaldehyde.

[c31] 31. The process of claim 30, wherein a sufficient quantity of said acetaldehyde is removed from said volatile phase to maintain a concentration of less than about 400 parts per million by weight of propionic acid in said purified acetic acid product.

[c32] 32. The process of claim 30, wherein a sufficient quantity

of said acetaldehyde is removed from said volatile phase to maintain a concentration of less than about 250 parts per million by weight of propionic acid in said purified acetic acid product.

[c33] 33.The process of claim 29, further comprising recycling at least a portion of the extracted PRC enriched overhead directly or indirectly to the carbonylation reactor.

[c34] 34.The process of claim 33, further comprising the step of adding dimethyl ether to the PRC enriched overhead.

[c35] 35.The process of claim 33, further comprising forming dimethyl ether during the distillation of said mixture and reacting at least a portion of the dimethyl ether with carbon monoxide in the reactor.

[c36] 36.The process of claim 35, further comprising the step of adding water to the mixture, to a stream associated with the distillation of the mixture, or into the first portion of the extracted PRC enriched overhead to promote formation of dimethyl ether during the distillation.